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PREPARATION OF CHEMICAL SAMPLES ON RELEVANT SURFACES USING INKJET TECHNOLOGY

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14. ABSTRACT
The Direct Jet 1309 printer (Direct Color Systems, Rocky Hill, CT) was used to generate the modeled distribution of actual chemicals on relevant surfaces. Various chemical simulant characteristics were evaluated for printing suitability. These characteristics included viscosity, surface tension, density, substrate surface energy, and harmfulness to the printer. Quantitative analyses were performed on printed materials. Results showed that the printer produced uniform distributions as well as quantitatively accurate samples within 7% of the predicted amount. When samples were printed on a heated substrate, particles were much smaller and more evenly distributed than at room temperature.

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PREFACE

The work described in this report was authorized under Defense Threat Reduction Agency project no. BA06DET018 and the Army Technology Objective on Detection of Unknown Bulk Explosives. The work was started in June 2010 and completed September 2012.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

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CONTENTS

FIGURES

TABLES

PREPARATION OF CHEMICAL SAMPLES ON RELEVANT SURFACES USING INKJET TECHNOLOGY

1. INTRODUCTION

The U.S. Army has a critical need to remotely detect chemical, biological, and explosive materials on surfaces. The detection of contamination on surfaces is a difficult and potentially costly problem to solve. To mitigate these risks, the personnel at the U.S. Army Edgewood Chemical Biological Center (ECBC) have been developing a predictive model for standoff detection of chemicals on surfaces. The model can be used to compare existing technologies as well as predict performances and identify system weaknesses. Currently, a generic Raman-based standoff detection system is being modeled and validated using laboratory measurements. The reliability of the model is based on its ability to reproduce a realistic contamination condition in the form of a synthetic witness card, and its ability to accurately simulate the pixel-to-droplet interaction. To validate the modeling, one aspect of the experimental setup must have the ability to fabricate witness cards in a controlled and uniform fashion.

In this work, the Direct Jet 1309 printer (Direct Color Systems, Rocky Hill, CT) was acquired to generate the modeled distribution on relevant surfaces with actual chemicals. In addition to modeling agent stimulants, the printer has been used to deposit explosive materials on relevant surfaces for the calibration of standoff explosive systems.

2. EXPERIMENT SETUP

2.1 Inkjet Printer

The Direct Jet 1309 flat-bed inkjet printer (Figure 1) is presently used to deposit various chemicals on surfaces, such as substrates of aluminum and Teflon as well as microscope slides. Concentrations of substrates from 1 to 100 μg/cm² are deposited in a single pass, and increased amounts use multiple coatings.

The following are characteristics of the Direct Jet 1309 printer:

- Prints directly on concrete, metal, glass, plastic, and so on;
- Maximum substrate size, 13 × 9 × 2 in.;
- Maximum substrate weight, 10 lb;
- Resolution range, 720 to 5760 dpi;
- Droplet size, 1.5 to 21 pL; and
- Print head hole diameter, approximately 23 μm.

Figure 1. Direct Jet 1309 inkjet printer.

The Direct Jet 1309 inkjet color printer is based on an Epson design using Epson-style cartridges and an Epson printer. The printer is supplied with eight empty ink cartridges that may be filled with ink, or in this case, chemicals to be deposited. Figure 2 and Table 1 show the typical ink colors and corresponding cartridges for that channel in the print head. By replacing the inks with chemicals and solvents, the printing system operates as a normal piezoelectric printer.

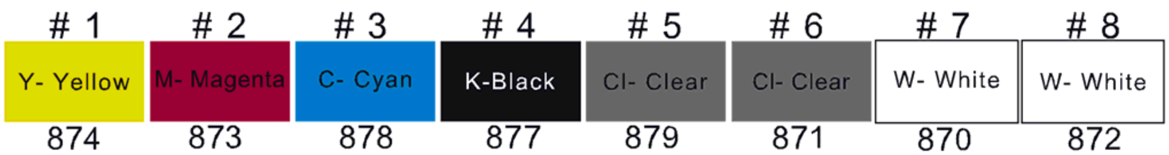


Figure 2. Typical ink colors and cartridges.

Table 1. Ink Cartridges

Channel Number	**Ink Color**	**Code on Cartridge**
1	Yellow (Y)	874
2	Magenta (M)	873
3	Cyan (C)	878
4	Black (K)	877
5	Clear (Cl)	879
6	Clear (Cl)	871
7	White (W)	870
8	White (W)	872

The printer resolution ranges from 720 × 720 to 1440 × 5760 dpi, and the droplet size ranges from 1.5 to 21 pL (although droplet size may vary with resolution). For initial experiments, a resolution of 720 × 720 dpi and a droplet size of 21 pL were programmed. The selected ink cartridge was filled with the selected chemical (referred to as the “color” in this report), and the other cartridges were filled with chemical solvent. All eight cartridges must be filled to avoid damage to the print head. To approximately double the amount of chemical deposited, two cartridges may be filled with the same chemical and 100% of both selected “colors” may be programmed. Recent printing experiments have used droplet sizes from 1.5 to 21 pL and a printer resolution of 1440 × 1440 dpi with excellent results. Because droplet size may be affected by chemical viscosity and surface tension, the actual droplet volume is determined experimentally.

2.2 Printer Software

The software programs provided with the printer are Color Byte Rip Pro and Color Byte Rip Pro Queue. A compiled summary of the printer operating procedure is included in Appendix A and summarized in Sections 2.3 and 2.4. When applying the chemicals using the printer, usually only one ink “color” is used. In the Color Byte software, cartridges are selected by the designated color, rather than by number. To print using selected ink cartridges, the software must specify the color as yellow, magenta, cyan, or black for ink cartridges 1 through 4, which are in turn coded to the particular chemical analyte to be deposited (i.e., cyan is actually ammonium nitrate). The cyan, magenta, yellow, and black (CMYK) system has 256 unitless intensities, from 0 to 255, for each color. Table 2 shows the intensity required for each CMYK color to produce the corresponding RGB color. The relationship between the red, green, and blue (RGB) and CMYK color systems is shown below.

Table 2. RGB/CMYK Intensity Relationship

	C	M	Y	K
R	0	255	255	0
G	255	0	255	0
B	255	255	0	0

Predesigned print patterns or images may be imported as a Joint Photographics Experts Group (jpg), bitmap, or vector graphics file. Prior to import, all file images should be of the same color, either yellow, magenta, cyan, or black.

2.3 Suitable Liquids

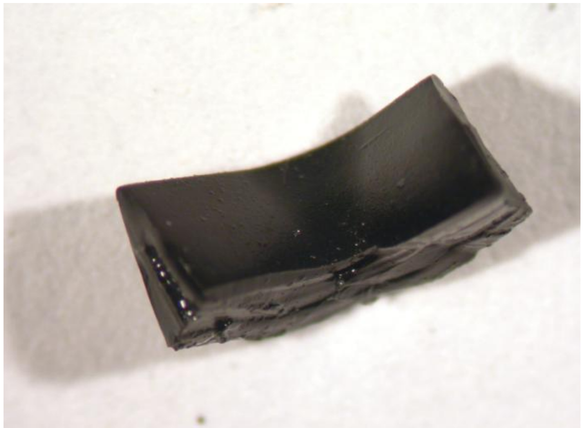
The Ink target viscosity range is from 1.5 to 5 centipoise (cP). This is the usual viscosity range for printer inks and provides a good range for printing chemicals. To print powder chemicals, a number of solvents have been used, including ethanol, acetonitrile, water, and water–alcohol mixtures. Viscosities up to 5 cP have been printed easily; however, surface tension has been of greater concern than viscosity, particularly for water solutions. The surface tension of most water-based inks is 34–40 dyn/cm. If the surface tension is too high, the ink may not wet nor travel through the ink cartridge correctly, thus causing an incorrect deposition. The pure water surface tension is 72.8 dyn/cm. This high surface tension prevents water from passing through some holes in the print head, which results in missed printed lines, insufficient solute volume, and in some cases, the absence of the print (material deposition) entirely. The addition of a small amount of surfactant (e.g., Tween 20 or 0.05% Tween-to-water) to the water completely eliminated this problem. As of the date of this report, analysis of the generic-based spectroscopic technique (Raman) used to evaluate surface depositions had not revealed any spectral interference due to the Tween 20. It should be noted that to prevent blocking the fine holes in the print head, all printing liquids were filtered to 1.0 µm.

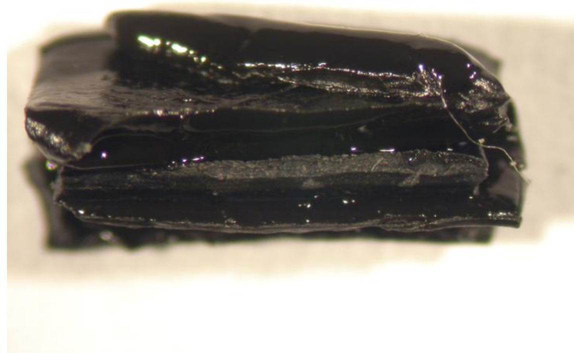
For initial printer calibration and to determine repeatability, a mineral oil viscosity standard was substituted for ink. Mineral oil was selected only because of its viscosity, and it was not considered an agent simulant. The mineral oil viscosity at 68 °F is 4.021 cP and the density is 0.8704 g/mL. Mineral oil performed well in the printing function, but a number of printer failures were caused when mineral oil attacked the rubber gasket in the printer capping station. These printer failures were initially attributed to printer manufacturing defects, so mineral oil was used extensively in early experiments. Printer capping station failures resulted in printer disablement but did not affect print quality. The capping station mechanism is in two sections, and each section covers four ink cartridges. Because mineral oil was used in only one ink cartridge, the rubber gasket on the second side was not damaged. Subsequently, the undamaged rubber gasket was cut up and each piece was soaked in mineral oil, ethanol, DCS solvent wash (Direct Color Systems), and SF96-5 (Momentive Specialty Chemicals, Columbus, OH) for 24 h. The microscope photographs in Figure 3 show damage caused by the mineral oil; other chemicals caused no damage.

Following the discovery that mineral oil has a negative effect on the printer’s internal parts, SF96 was chosen for repeatability measurements. SF96-5 is a low-surface-tension polydimethylsiloxane fluid, commonly used as a base fluid in personal care products. It is a clear liquid with a distinct, recognizable Raman signature that also makes it a suitable liquid chemical agent simulant. SF96-5 has a viscosity of 5 cP, which is comparable to printer ink. The disadvantage however, is its low surface tension of 19.7 dyn/cm, which causes complete wetting on high-surface, free-energy materials such as metal and glass. However, a suitable substrate material is polytetrafluoroethylene (PTFE [Teflon, E.I. DuPont de Nemours, Wilmington, DE]) on which printed patterns of SF96-5 showed very little spreading.

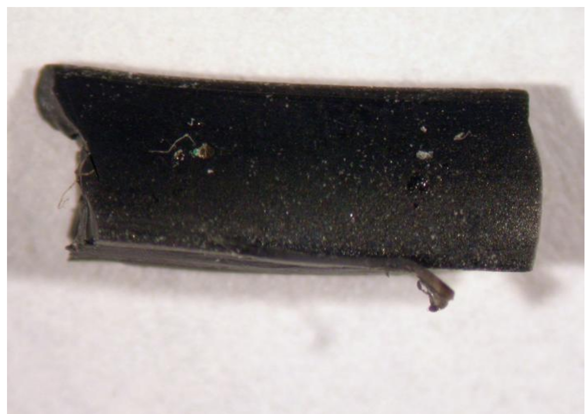
Specifications for SF96-5 and Teflon at 25 °C were as follows:

- SF96-5 specific gravity: 0.913
- SF96-5 surface tension: 19.7 dyn/cm
- SF96-5 viscosity: 5.0 cP
- Teflon surface free energy: 20.0 dyn/cm





SF96-5 Mineral Oil



Ethanol Solvent Wash

Figure 3. Effect of various chemicals on rubber gaskets.

2.4 Print Procedure

A number of tests are required to determine how various printer settings and chemical parameters affect the chemical mass deposited. Printer settings include resolution, drop size, drop volume, number of coats, maximum ink, and number of print cartridges used. Chemical parameters include viscosity, surface tension, and density. Chemicals may be liquid or a solid dissolved in a suitable solvent. The volume per droplet may be specified in the printing program as 7, 14, or 21 pL; however, these volumes were determined using printer ink and were not necessarily accurate for the chemicals to be deposited and had to be reevaluated.

Determination of actual chemical mass deposited required a number of steps. Initially, the substrate was to be weighed before and after chemical deposition. In practice, however, this procedure is not always practical because the anticipated chemical mass may be in the milligram or microgram range, and the background substrate may be weighed in grams. In these cases, the resolution of the available laboratory scale may provide inadequate results. Initially, the procedure used for determining the chemical mass deposited on a heavy substrate is to use a small, lightweight aluminum substrate to determine the droplet volume. This may be accomplished by weighing the substrate before printing and again after the substrate is dried using a heat gun. With knowledge of the chemical concentration of the liquid, the droplet volume may be calculated. Some chemicals used for printing, such as ammonium nitrate, are very hygroscopic, and care must be exercised when weighing the substrate. Weight will initially decrease as the substrate is dried. The weight then increases as moisture is absorbed by the chemical from the air. The weight eventually stabilizes as the chemical moisture content approaches that of the air. This stabilized weight measurement probably introduces the least experimental error by assuming that the chemical moisture content at the time of this measurement is similar to the moisture content when the chemical was first dissolved.

Equations 1 through 4 are useful chemical deposition formulas. The constant *K*, which appears dimensionless, was derived to simplify calculations by allowing numerical values to be used for droplets in picoliters, area in square centimeters, and concentrations in grams per liter. The value of *K* is determined by droplet area, which varies with printer resolution or dots per inch (dpi).

$$C = (S \times K)/P \tag{1}$$

$$P = (K \times M)/(A \times C) \tag{2}$$

$$M = (P \times A \times C)/K \tag{3}$$

$$S = M/A \tag{4}$$

where

A is surface area (cm^2);
P is droplet (pL);
C is liquid concentration (g/L);
K is 9.775E+6 (for 720 dpi), K is 2.44E+6 (for 1440 dpi);
M is mass deposited (g); and
S is surface concentration (g/cm^2).

3. PRINT MODES

Various print modes were tested for ways to vary the material concentrations on surfaces. Density of materials can be controlled by changing the variable dot size (VDS) and/or dots per inch (dpi) settings in the printer.

For the results in Section 3.1, 32 g/L of potassium chlorate was printed on a 2 × 2 in. pattern of aluminum substrate. All of the printed samples were heated to 80 °C. The printer setting combinations were 1.5, 3, 7, 14, and 21 pL for the VDSs and 720 × 720 and 1440 × 1440 dpi. All the samples were weighed after deposition.

3.1 Results

The following results (Table 3) were obtained during testing:

- It was determined that the “Small Dots” VDS settings should not be used because the printer did not deposit consistent mass on the surface. This result was labeled as “RED”, not usable.
- 3 pL setting: Did not print all the way with heating pad but worked fine without the heat. Assumption is that printer head holes were dried by the heat and became clogged. This result was labeled as “YELLOW”, can be used with caution.
- 7 pL setting: Worked the best with VDS2, all medium dots. This result was labeled as “GREEN”, usable.
- 14 pL setting: Worked the best with VDS1, all medium dots. This result was labeled as “GREEN”, usable.
- 21 pL setting: Worked best with VDS1, all large dots. This result was labeled as “GREEN”, usable.

Table 3. Print Mode vs Dot Sizes

Dot Size	VDS1 (pL)	VDS2 (pL)	VDS3 (pL)
Small	7	3	1.5
Medium	14	7	3
Large	21	14	7

The print mode files used in this study were set up with these parameters: resolution in dots per inch, International Color Consortium (ICC) setting (where “No ICC” is the setting used for 100% color of ink cartridge), unidirectional printing (uni) setting, and IR3 ink type. The following print mode files were used:

720 × 720 dpi, IR3, No ICC, 21 pL, uni
1440 × 1440 dpi, IR3, No ICC, 21 pL, uni

720 × 720 dpi, IR3, No ICC, 14 pL, VDS1, uni
1440 × 1440 dpi, IR3, No ICC, 14 pL, VDS1, uni

720 × 720 dpi, IR3, No ICC, 7 pL, VDS2, uni
1440 × 1440, IR3, No ICC, 7 pL, VDS2, uni

720 × 720 dpi, IR3, No ICC, 3 pL, VDS3, uni
1440 × 1440 dpi, IR3, No ICC, 3 pL, VDS3, uni

Table 4. Validation of Picoliters per Droplets

Print Modes Measured			
Dots per Inch	Picoliters	Mass	Measured Picoliters
720 × 720, uni	1.5	90 µg	1.07
720 × 720, VSD3, uni	3	260 µg	3.08
720 × 720, VSD2, uni	7	690 µg	8.17
720 × 720, VSD1, uni	14	1.34 mg	15.86
720 × 720	21	2.11 mg	24.97
1440 × 1440, VSD2, uni, 80 °C	7	2.68 mg	7.93
1440 × 1440, VSD1, uni, 80 °C	14	5.09 mg	15.06
1440 × 1440, 80 °C	21	7.74 mg	22.90

4. QUANTIFICATION ANALYSIS

4.1 Energetic Print Testing of 3 × 3 in. Aluminum Panels

For ground truth, the density of coupon deposition was verified using several methods including theoretical calculation, coupon mass measurement, and chemical laboratory analysis.

A 2 × 2 in. pattern of ammonium perchlorate (APC) was printed on 3 × 3 in. aluminum panels (Figure 4).

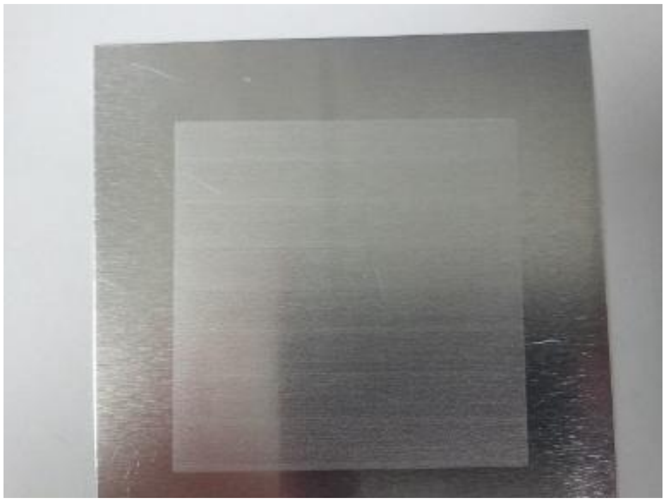


Figure 4. The 3 × 3 in. aluminum sample panel.

4.1.1 Calculation

The printed mass is calculated by first determining the solution concentration for the desired deposition. For example, for a 50 μg/cm^2 surface concentration, eq 5 is used calculate a solution concentration (grams per liter)

$$C = (S \times K)/P \tag{5}$$

where

S is 50E–6 g/cm^2,
K is 9.775E+6 (720 DPI),
P is 14 pL/drop, and
C is 34 g/L.

The total printed mass is then calculated using eq 6

$$M = (P \times A \times C)/K \tag{6}$$

where

P is 14 pL/drop,
A is 25.81 cm^2,
C is 32 g/L,
K is 9.775E+6 (720 dpi), and
M is 1.2 mg.

4.1.2 Coupon Mass Measurement

Samples were printed using the following options: 720 × 720 dpi, 14 pL/drop, and bidirectional printing. The total printed mass is measured by subtracting the mass of the blank aluminum panel from the mass of the panel with the printed sample.

4.1.3 Verification

Table 5 shows the results for all samples that were sent to the laboratory for verification using the extraction method described in Section 4.1.4. The overall average difference of the verification results (VR) when compared with the coupon mass measurement was only 7%.

Table 5. Comparison of VR to Measured and Calculated Values

Sample Description	Sample Size (µg/cm^2)	Calculated Mass (mg)	Printed Amount Measured (mg)	VR (mg)	|VR – Measured| /VR (%)
APC-1020-1	50	1.29	1.41	1.65	15
APC-1020-2	65	1.68	1.67	1.60	5
APC-1020-3	59	1.52	1.53	1.56	2
APC-1028-1	54	1.39	1.39	1.38	1
APC-1028-2	47	1.21	1.21	1.39	13
APC-1028-3	50	1.29	1.30	1.21	7
APC-1028-4	60	1.55	1.57	1.21	29
APC-1031-101-1	101	2.61	2.61	2.79	7
APC-1031-101-2	101	2.61	2.61	2.83	8
APC-1103-53-1	53	1.37	1.37	1.35	1
APC-1103-100-1	100	2.58	2.60	3.09	16
APC-1102-51-1	51	1.32	1.31	1.34	2
APC-1102-49-2	49	1.26	1.27	1.39	9
APC-1102-111-3	111	2.86	2.87	2.92	2
				Average	7

4.1.4 APC Extraction Method

Five Ziploc brand, plastic polypropylene (PP) containers were obtained and rinsed by pipetting 40 mL of deionized (DI) water into the containers. The surface of each aluminum panel was rinsed ~25 times by adding the same solvent (DI water) into the containers until no APC could be seen on the panel surface. The aluminum panels were then placed face down into the plastic containers, and the lids were snapped on. All five containers were then placed onto an orbital shaker table and allowed to shake at a low speed (~100 rpm) for ~18 h.

A 500 µL aliquot of sample was removed from each plastic container using an automatic pipette (set at 500 µL). The samples were placed into separate, labeled, plastic autosampler vials and submitted for ion chromatography (IC)–conductivity detection (CD) analysis. Most of the remaining sample extracts (~20.0 mL) were transferred to 20 mL plastic bottles and stored in the refrigerator at ~8 °C.

The following information was used for the calculations in this study:

- APC (NH_4ClO_4) = 117.493 g/mol
- Ammonium (NH_4^+) = 18.042 g/mol [15.36% of APC]
- Perchlorate (ClO_4^-) = 99.451 g/mol [84.64% of APC]

4.1.5 Depositing Actual Chemicals on Relevant Surfaces

In this test, five 10 mm diameter round spots were printed on a 2 × 2 in. Teflon substrate. The test pattern is shown in Figure 5.

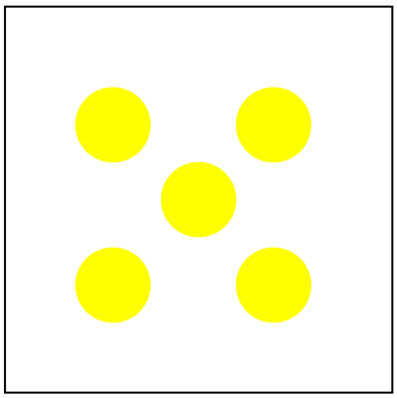


Figure 5. A 2 × 2 in. test pattern.

Table 6. Chemical Deposition Results on Teflon Using Yellow Cartridge

Sample Number	Weight Before Printing (g)	Weight After Printing (g)	Difference (mg)
1	4.79859	4.80206	3.47
2	4.81485	4.81835	3.50

The following results were also obtained:

- Mean weight difference: 3.49 mg
- Total area of five 10 mm spots: 392.7 mm²
- At 720 × 720 dpi resolution: 518,400 dots/in.² = 803.52 dots/mm²
- Total dots: 803.52 × 392.7 = 315,540 dots
- Weight per dot: 3.49E–3/315.50E+3 = 11.062E–9 g
- SF96-5 density: 913 g/L
- Volume per droplet: 11.06E–9/913 = 12.1 pL

This measurement was repeated using SF96-5 in printer cartridge #2 (magenta).

Table 7. Chemical Deposition Results on Teflon Using Magenta Cartridge

Sample Number	Weight Before Printing (g)	Weight After Printing (g)	Difference (mg)
1	4.79959	4.80354	3.95
2	4.81442	4.81828	3.86

The following results were also obtained:

- Mean weight difference: 3.91 mg
- Total area of five 10 mm spots: 392.7 mm²
- At 720 × 720 dpi resolution: 518,400 dots/in.² = 803.52 dots/mm²
- Total dots: 315,540
- Weight per dot: 3.91E–3/315.50E+3 = 12.393E–9 g
- Volume per droplet: 12.393E–9/913 = 13.57 pL

These experimental measurements of droplet volume were significantly less than 21 pL per droplet, which was stated in the printer specification. However, the printer manufacturer did not provide the conditions needed for using 21 pL per droplet. To possibly increase the droplet volume, an experiment was conducted wherein the yellow and the magenta ink cartridges were both used, and each cartridge was set to 100%.

Table 8. Chemical Deposition Results on Teflon Using Yellow and Magenta Cartridges

Sample Number	Weight Before Printing (g)	Weight After Printing (g)	Difference (mg)
1	4.79950	4.80773	8.23
2	4.81850	4.82709	8.59

The following results were also obtained:

- Mean weight difference: 8.41 mg
- Volume per droplet: 29.2 pL

4.2 Vapor, Liquid, and Solid Tracking (VLSTRACK) Witness Card

The data obtained from using VLSTRACK for testing provided a mass of 20.1 mg of SF96-5 on a 6 × 6 in. witness card. The pixel count was 176,980. To calculate the number of coats of SF96-5 required to print 20.1 mg of material, the following parameters were used:

- Mass per droplet: 913 × 29.2E–12 = 26.7E–9 g
- Mass per coat: 176,980 × 26.7E–9 = 4.725 mg per coat

- Number of coats: 20.1/4.725 = 4.25 coats
- Weight of 6 × 6 in. Teflon substrate before printing: 61.0356 g
- Weight after four coats: 61.05538 g
- Weight difference: 19.78 mg

This result meets the desired printed target of 20.1 mg of SF96-5 within 1.6%.

5. PRINTING ON A HEATED SUBSTRATE

When printing aqueous solutions of chemicals with the Direct Jet 1309 printer, the dried chemical crystal residue patterns exhibit significant voids. Although printing may be set at 720 dpi, the crystal patterns may be as sparse as 150 crystals per inch. This may be attributed to the fact that the printer droplets do not dry instantly as they hit the substrate, and the puddles that form join together to grow larger crystals as the solvent dries. It has been observed that when similar chemical solutions are paint-sprayed onto a hot (70 °C) substrate, the crystals are smaller, more numerous, and more closely spaced. To facilitate printing on heated substrates, a temperature-controlled hotplate was designed and installed on the printer bed. Substrate temperature is maintained at 80 °C. Figures 6 and 7 are photographs of potassium chlorate crystals deposited with and without heating. Both photographs are at the same scale.

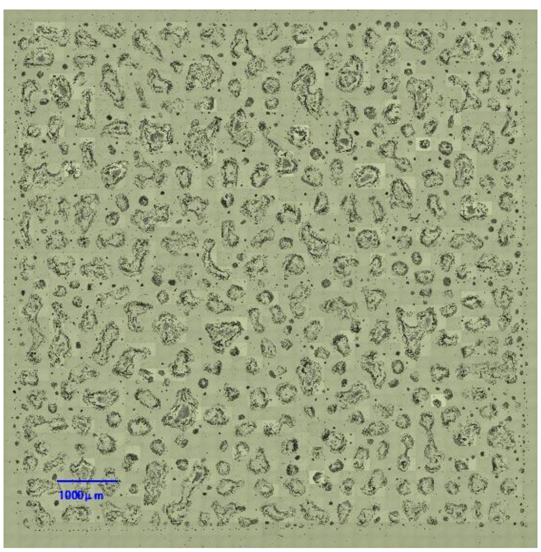


Figure 6. $KClO_3$, no heating, 54 µg/cm².

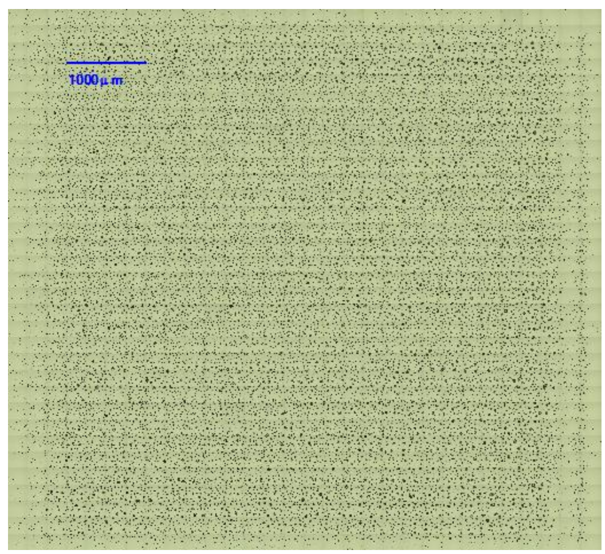


Figure 7. $KClO_3$, heated to 80 °C, 45 µg/cm².

5.1 Heater Design

The Direct Jet 1309 printer allows a substrate height of 2 in., a maximum substrate size of 13 × 10 in., and a maximum weight of 10 lb. Sufficient space was provided for the following heater configuration: A 10 × 10 in. block of 1¼ in. PP was mounted on the printer table using the existing tacky mat. A 0.10 in. thick, flexible Kapton heater was adhesive-mounted on top of the PP block. Because PP has a thermal conductivity of 0.10–0.22 W/mK and a working temperature of 90–120 °C, thermal insulation is provided for the heater. The metal printer table is considered an infinite thermal heat sink at ambient temperature. A tacky mat, similar to the printer table mat, was placed on top of the heater. A thermocouple was placed between the tacky mat and the heater for the measurement and control of substrate temperature. The thin tacky mat was assumed to be rubber with negligible thermal resistance. The tacky mat was tested in boiling water (100 °C) and had no apparent change in tacky characteristics due to temperature.

To calculate the PP thermal resistance and the power required to raise the substrate to 70 °C

$$\text{Thermal resistance, } R_\theta = l/(k \times A) \qquad (7)$$

where

k is thermal conductivity (W/mK),
l is thermal path length (material thickness [m]), and
A is area (m^2).

For PP:
$k = 0.15$ W/mK,
$l = 31.75E–3$ m,
$A = 64.5E–3$ m^2, and
$R_\theta = 3.28$ °C/W.

For an ambient temperature of 25 °C and a substrate temperature of 70 °C, ΔT is 45 °C; therefore, power (P) is calculated as

$$P = \Delta T/R_\theta = 13.7 \text{ W} \qquad (8)$$

This calculation considers only thermal conduction. Additional substrate cooling will be from convection as well as radiation. According to the *RCA Silicon Power Circuits Manual Technical Series Sp-50*, 2nd ed. (RCA Victor Company; Camden, NJ; 1967) the free air-convection thermal resistance of a vertically mounted 10 × 10 in. plate with a surface temperature of 70 °C at 25 °C ambient is 2.95 °C/W. A vertically mounted plate has approximately 30% less thermal resistance than a horizontal plate under the same thermal conditions, which means the resistance of the horizontal plate is approximately 4.2 °C/W. The net thermal resistance due to both conduction and convection is then approximately 1.84 °C/W, which requires a power of 24.5 W to maintain a temperature of 70 °C. The additional power required, due to thermal radiation, depends upon emissivity of the heated substrate surface, which will be determined by experiment. Sufficient additional power is available to account for cooling due to radiation.

An Omega Engineering (Stamford, CT) CN742 temperature controller with a solid-state relay was used to drive the heater, which was rated for 250 W at 115 Vac voltage to the heater from the alternating current (AC) mains was reduced by an electrically isolated Variac.

6. SUMMARY

Deposition of chemical samples on relevant surfaces using the Direct Jet 1309 flat-bed inkjet printer produced uniform distributions as well as quantitatively accurate samples within 7% of the predicted amounts. When samples were printed on a heated substrate, the particles were much smaller and more evenly distributed than when printed at room temperature.

Blank

ACRONYMS AND ABBREVIATIONS

AC	alternating current
APC	ammonium perchlorate
CD	conductivity detection
CMYK	cyan, magenta, yellow, and black
cP	centipoise
DI	deionized (water)
dpi	drops per inch
ECBC	U.S. Army Edgewood Chemical Biological Center
IC	ion chromatography
ICC	International Color Consortium
jpg	Joint Photographics Experts Group (file extension for graphics)
LED	light-emitting diode
PP	polypropylene
PTFE	polytetrafluoroethylene
RGB	red, green, and blue
uni	unidirectional printing
VDS	variable dot size
VLSTRACK	vapor, liquid, and solid tracking
VR	verification results

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APPENDIX A
PRINTER INSTRUCTIONS FOR THE DIRECT JET 1309

A.1 TO CHANGE THE INK CARTRIDGES

Follow these steps to remove existing ink cartridges and install new ones:

1. Turn main power switch on. Verify that the amber light on green button is lit.
2. Press the green button. The print engine will lift approximately 1 in. Wait until the light-emitting diode (LED) is solid green.
3. Press the Media Home button.
4. Press the blue button. The print station will move to the load position.
5. Remove the existing cartridges and install the new cartridges.
6. Press the blue button again. The carriage will move to the capping position and air will be purged. Wait until the LED is solid green.
7. Press and hold the blue button until the green LED starts flashing. The printer will perform the head-cleaning procedure. Wait until the LED is solid green. Repeat step 6. (Perform another head cleaning.)
8. Perform the nozzle-check pattern (Section A.2). If required, repeat step 6.

A.2 TO PERFORM NOZZLE CHECK PATTERN

Follow these steps to check the nozzle pattern:

1. Ensure that the green button LED is solid green. (If light is amber, press green button.)
2. Press the Media Home button. (The table will go to Media Home position.)
3. Place plain white paper on the table at top left corner. Press firmly on the tacky mat.
4. Press the Print Home button.
5. Press and hold the down arrow until the print engine stops.
6. Open the Color Byte Rip Pro Queue software program.
7. Check OK on Ink Pad Reset.
8. Select Queue from the toolbar then choose Manage Queue.
9. Under Port, select Epson Stylus Pro then choose Close.
10. Click on Printer Status then on the Settings icon.
11. Click on Nozzle Check Pattern.

A.3 LOADING SUBSTRATE

Follow these steps to load the substrate:

1. Turn the main power switch on. Verify the amber light on the green button is lit.
2. Press the green button. The print engine will lift approximately 1 in., and the table will move. Wait until the LED is solid green.
3. Depress the Media Home button.
4. Press and hold the up arrow until there is enough space between the table and print engine cover to clear the substrate that will be printed on.
5. For paper, place the paper at top left corner of the tacky mat. Press firmly on the tacky mat. For other materials, place the substrate at top left corner using guides.
6. Physically push the media table toward the print engine, making sure that the substrate material clears the bottom edge of the print engine cover. Push the media table approximately 2 in. past the edge of the print engine cover. The height sensor will adjust the print engine to the proper height. If the print engine does not move, you have started too high. Try again in a lower position.
7. Depress Print Home button; you are now ready to print. (All buttons are now locked. To release lock, simultaneously press the right arrow and Media Home button.)

A.4 HEAD-CLEANING PROCEDURE

Follow these steps to clean the print head:

1. Turn the main power switch on. Verify the amber light on the green button is lit.
2. Press the green button. (The print engine will lift approximately 1 in. and the table will move. Wait until the LED is solid green.
3. Press the Media Home button.
4. Press and hold the blue button until the green LED starts flashing. (The printer will perform the head-cleaning procedure.)
5. Wait until the green LED lights up solid green before starting any other procedures.

A.5 PRINTING WITH CHEMICALS

Follow these steps to print with chemicals:

1. Open the RIP PRO software.
2. Select LAYOUT from toolbar.
3. In the BLANK SIZE field, input the substrate size, check Portrait. Click OK.

4. Double click on the black SF square.
5. Select CMYK. Change C, M, Y, or K to 100, change all others to 0.
6. Select Process.
7. Input the color name (experiment name).
8. Click Change.
9. Load the image on screen using Import or the Select Shape tool.
10. Place an angle in upper left corner (if required).
11. Choose Save as.
12. Select File/Print & Cut.
13. Click on box with three dots next to Printer Name.
14. Click on box with three dots next to Print Mode.
15. Select the Print Mode, example: 720 × 720 IR3 No ICC Norm. Click OK. (See the section on Changing Print Mode.)
16. Click on the Page Setup tab. Select Custom then Portrait and check the height and width.
17. Click on the Preferences tab. Select Sign Blank Area, bitmaps, verify number of coats, and copies then click OK.
18. Click on the printer icon in Print/Cut toolbox.
19. In the Spot Foil Browser, click OK.
20. Right click on the job and rename it.
21. For multiple coats, press the yellow Auto-return button, right click on Properties, and in Printer Options, check Change Default Printer Options. Change the End Delay to 50 seconds.
22. Right click on Properties then verify the Dot Setup and Max Ink Settings.
23. Right click on Print.

A.6 CHANGING PRINT MODE

The print mode may be modified to change the ratio of small, medium, and large dots (the default setting is all large dots) or to change the picoliters per dot. Dot volume settings in picoliters are shown on the following chart. Procedure steps are shown below.

1. Launch the Rip Pro Software.
2. Select File/ Launch Visual Production Manager.
3. Rename, edit, and make changes on copy.
4. Right click on Print Mode to modify.
5. Click on copy.
6. Rename, edit, and make changes to copy.

Dot Size	VDS1 (pL)	VDS2 (pL)	VDS3 (pL)
Small	7	3	1.5
Medium	14	7	3
Large	21	14	7

Blank

APPENDIX B
PATENT APPLICATION

DAM 757-12: Device and Method for Precisely Depositing Chemical/Biological Materials on Surfaces

B.1 BACKGROUND

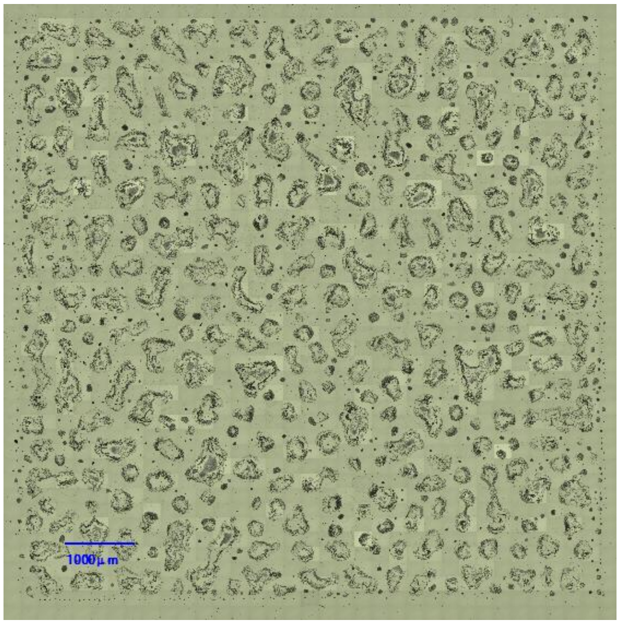
Materials deposited on manmade or natural surfaces are frequently used as calibration standards for chemical- or biological-detecting systems. To determine the threshold sensitivity of systems that detect explosives and chemical agents, precise and accurate quantities of contaminants must be deposited. Present drop-and-dry deposition systems produce a largely nonuniform distribution of particles with an overall sample spread shape and area that is difficult to control or predict. This invention allows precise control of droplet volume, droplet size, and dots per inch, for accurate material deposition.

B.2 SUMMARY OF INVENTION

This invention is a process for depositing chemical and/or biological materials on natural or manmade surfaces in precisely controlled quantities and patterns. The process utilizes a commercial flat-bed inkjet printer modified to include a temperature-controlled heated substrate area. Printer ink cartridges are filled with the material to be deposited, in liquid form, consistent with normal printer ink specifications regarding specific gravity, surface tension, particle size, and chemical composition. Substrate material may be wood, metal, glass, plastic, concrete, road blacktop, dirt, or other suitable materials.

When printing aqueous solutions of chemicals at ambient temperature, the dried chemical crystal residue patterns may exhibit significant voids. Although printing may be at 720 dpi, the crystal patterns may be as sparse as 150 crystals per inch. This may be attributed to the fact that the printer droplets do not dry instantly as they hit the substrate, and the puddles that form join together to grow larger crystals as the solvent dries. The modified printer of this invention utilizes an optional temperature-controlled heated printer bed which, when activated, will heat the substrate to a typical temperature of 85 °C. Utilizing this heated substrate, chemical droplets dry instantly, resulting in a separate crystal residue for each droplet printed. This invention is the only known method of printing uniform patterns of chemical crystals in the submicron size range.

Figures B-1 and B-2 shows potassium chlorate crystals deposited with and without heating. Both photographs are at the same scale.



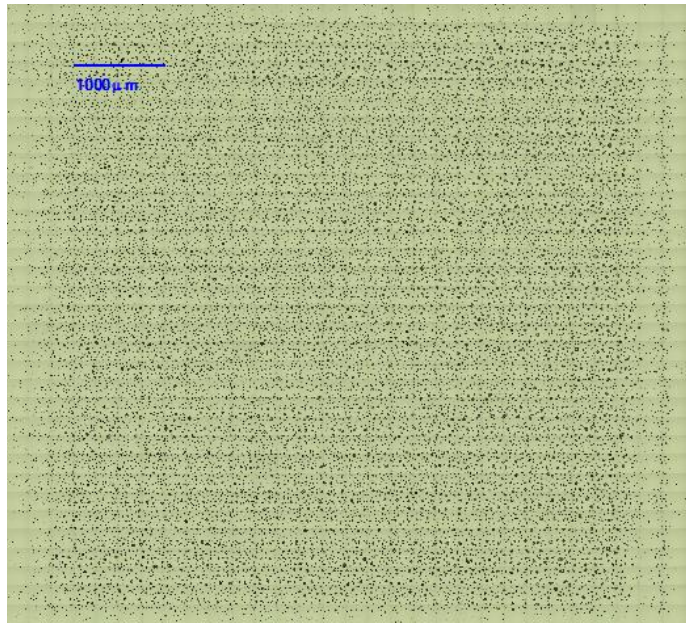


Figure B-1. $KClO_3$, no heating, 54 µg/cm².

Figure B-2. $KClO_3$, heated to 80 °C, 45µg/cm².

Another advantage of this invention is the ability to separate polymorphic crystal phases in materials such as the explosive RDX. Different crystal phases are observed at different concentrations. An accurate and controlled sample preparation technique is necessary to calibrate and evaluate optical detection technologies for the polymorphic analyte. The use of the inkjet printer satisfies this requirement. Figure B-3 shows the results of a concentration of 30 mg/mL RDX in acetonitrile applied with 40 successive printer coatings to a microscope slide substrate. A single coat showed essentially all β-RDX. Successive coats increased the ratio of α-RDX to β-RDX until the 40 coats were essentially all α-RDX. Figure B-3 shows the results of this test.

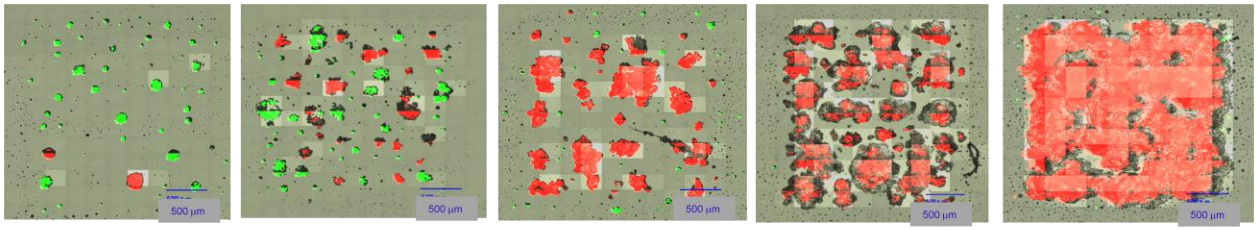


Figure B-3. Raman chemical images of RDX samples printed on an aluminum-coated microscope slide. Green indicates the presence of β-RDX and red indicates α-RDX. Different numbers of coatings, ranging from 1 to 40, were printed on different slides to illustrate the transformation from β-RDX to α-RDX.

Application of this invention can extend to the deposition of biological materials. The ability to print surface depositions in a range from 1 to 100 µg/cm² may prove applicable to the study of basic mechanisms governing selective binding of biomolecules with diverse substrate surfaces. Because the diameter of the hole in a typical print head is in the order of 20 µm, and bacteria diameters are in the 1 to 2 µm range, the deposition of bacteriological material appears practical.

B.3 PREFERRED EMBODIMENT

The Direct Jet 1309 flat-bed inkjet printer is presently used to implement this invention. Concentrations from 1 to 100 µg/cm² are deposited in a single pass with increased amounts using multiple coatings.

The following are some pertinent characteristics of the Direct Jet 1309 printer:

- Prints directly on concrete, metal, glass, plastic, and so on;
- Maximum substrate size, 13 × 9 × 2 in.;
- Maximum substrate weight, 10 lb;
- Resolution range from 720 to 5760 dpi; and
- Droplet size from 1.5 to 21 pL.

Figure 4 is a photograph of the Direct Jet 1309 flat-bed printer modified by the addition of a substrate heater mounted on the printer bed. A microscope slide to be printed is shown on the heater.

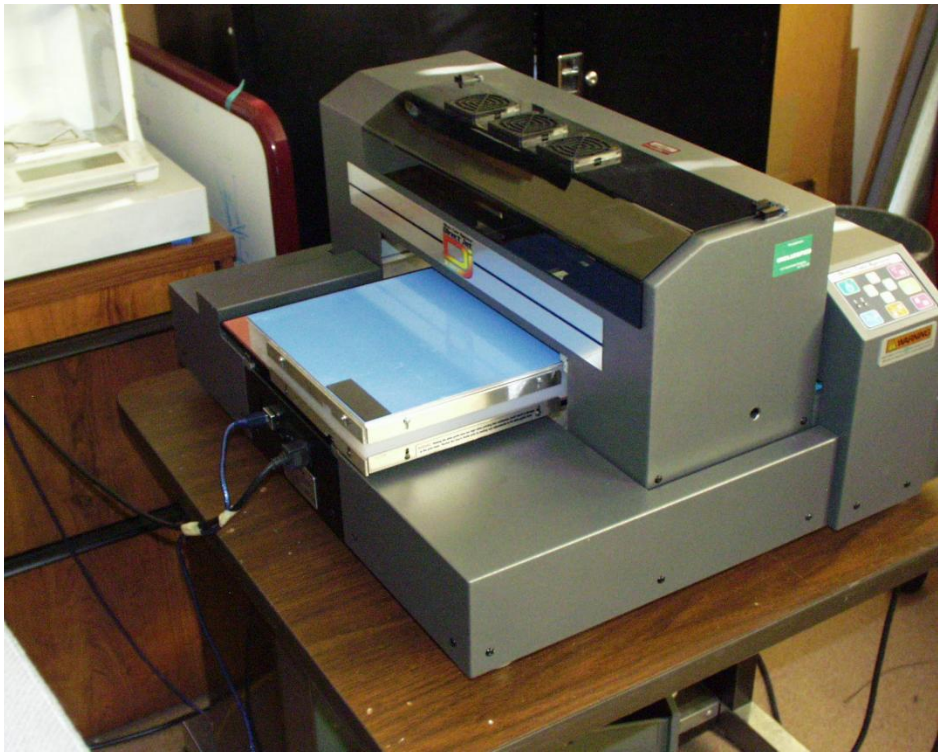


Figure B-4. Direct Jet 1309 inkjet printer.

The Direct Jet 1309 inkjet color printer is based on an Epson design. Epson style cartridges and an Epson print head are used. The printer is supplied with eight empty ink cartridges that may be filled with ink, or as in this case, filled with the chemicals to be deposited. Droplet size may vary with resolution. Advertised printer resolution ranges from 720 × 720 to 1440 × 5760 dpi and the droplet size ranges from 1.5 to 21 pL. Scanning may be bidirectional or unidirectional. For initial experiments, a resolution of 720 × 720 dpi and a droplet size of 21 pL were programmed. The selected ink cartridge is filled with the chemical to be printed and the other cartridges are filled with chemical solvent. All eight cartridges must be filled to avoid damage to the print head. To approximately double the amount of chemical deposited, two cartridges may be filled with the same chemical, and 100% of both selected "colors" may be programmed. Recent printing experiments have utilized droplet sizes from 1.5 to 21 pL and printer resolution to 1440 × 1440 dpi, with excellent results. Because droplet size may be affected by chemical viscosity and surface tension, the actual droplet volume is determined by experiment in critical experiments.

B.3.1 Heater Design

Sufficient space is provided for the following heater configuration. The Direct Jet 1309 printer allows a substrate height of 2 in., a maximum substrate size of 13 × 10 in., and a maximum weight of 10 lb. A 0.190 in. thick, 10 × 10 in. aluminum plate is mounted on top of a 10 × 10 in. block of 1¼ in. PP, which is mounted on the printer table using the existing tacky mat. A 0.10 in. thick, flexible Kapton heater and thermocouple are mounted between the aluminum plate and the PP block. Temperature control of the aluminum plate, in the range of 70 to 90 °C, is maintained by a commercial temperature controller, which is not considered part of the invention. Because PP has a thermal conductivity of 0.1–0.22 W/mK and a working temperature up to 120 °C, thermal insulation is provided for the heater. The metal printer table is considered an infinite thermal heat sink at ambient temperature. A tacky mat, similar to the printer table mat, is on top of the aluminum plate for mounting the substrate.

To calculate the PP thermal resistance and the power required to raise the substrate to 70 °C

$$\text{Thermal resistance, } R_\theta = l/(k \times A)$$

where

k is thermal conductivity (W/mK);
l is thermal path length (material thickness, m); and
A is area (m^2).

For PP:
$k = 0.15$ W/mK,
$l = 31.75\text{E}{-3}$ m,
$A = 64.5\text{E}{-3}\ \text{m}^2$, and
$R_\theta = 3.28$ °C/W.

For an ambient temperature of 25 °C and a substrate temperature of 70 °C, ΔT is 45 °C; therefore, power (P) is calculated as

$$P = \Delta T / R_\theta = 13.7 \text{ W}$$

This calculation considers only thermal conduction. Additional substrate cooling will be from convection as well as radiation. According to the *RCA Silicon Power Circuits Manual Technical Series Sp-50,* 2nd ed. (RCA Victor Company; Camden, NJ; 1967) the free-air convection thermal resistance of a vertically mounted 10 × 10 in. plate with a surface temperature of 70 °C at 25 °C ambient is 2.95 °C/W. A vertically mounted plate has approximately 30% less thermal resistance than a horizontal plate under the same thermal conditions, which puts this horizontal plate at approximately 4.2 °C/W. The net thermal resistance, due to both conduction and convection, is then approximately 1.84 °C/W, which requires a power of 24.5 W to maintain a temperature of 70 °C. The additional power required because of thermal radiation depends upon the emissivity of the heated substrate surface that will be determined by experiment. Sufficient additional power is available to account for cooling due to radiation.

B.3.2 Printer Software

The software programs provided with the printer are Color Byte Rip Pro and Color Byte Rip Pro Queue. In chemicals application, usually only one ink "color" is used. In the Color Byte software, cartridges are selected by the designated color, rather than by number. To print using selected ink cartridges, the software must specify the color as yellow, magenta, cyan or black for ink cartridges 1 through 4. The CMYK system has 256 intensities, from 0 to 255, for each color. Table B-1 shows the intensity of each CMYK color to produce the corresponding RGB color. The relationship between the RGB and CMYK color systems is shown below.

Table B-1. RGB/CMYK Intensity Relationship

	C	M	Y	K
R	0	255	255	0
G	255	0	255	0
B	255	255	0	0

Imported files may be jpg, bitmap, or vector graphics files. Prior to import, all file images should be of the same color, either yellow, magenta, cyan, or black.

B.3.3 Suitable Liquids

The ink target viscosity range is from 1.5 to 5 cP. This is the usual viscosity range for printer inks. A suitable liquid is SF96-5, which is a low-surface-tension polydimethylsiloxane fluid that is commonly used as a base fluid in personal care products. SF96 is a clear liquid with a distinct, recognizable Raman signature that makes it a suitable liquid chemical agent simulant.

The viscosity of SF96-5 is 5 cP, which is comparable to printer ink. One disadvantage in printing with SF96-5 is its low surface tension of 19.7 dyn/cm, which causes complete wetting on high-surface-tension, free-energy materials such as metal and glass. However, a suitable substrate material is PTFE (Teflon), on which printed patterns of SF96-5 show very little spreading. Specifications for SF96-5 and Teflon at 25 °C are as follows:

- SF96-5 specific gravity: 0.913
- SF96-5 surface tension: 19.7 dyn/cm
- SF96-5 viscosity: 5.0 cP
- Teflon surface free energy: 20.0 dyn/cm

To print dry chemicals, a number of solvents have been used. These solvents include ethanol, acetonitrile, water, and water–alcohol mix. Viscosities up to 5 cP have printed well; however, surface tension has proven to be of greater concern than viscosity. The surface tension of most water-based inks is 34–40 dyn/cm. If surface tension is too high, the ink may not wet or travel through the ink cartridge correctly.

The print-head hole diameter measures approximately 23 µm, which may also influence the passage of solvent through the cartridge. The use of water as a chemical solvent initially caused printing problems in the Direct Jet 1309 printer. Water surface tension is 72.8 dyn/cm, which is high enough to prevent water from passing through some holes in the print head, resulting in missed printed lines, insufficient solute volume, and, in some cases, the entire absence of any printing. The addition of a small amount of surfactant (Tween 20) to the water completely eliminated this problem. As little as 0.05% Tween-to-water mixture has proven sufficient. As of this date, the Raman chemical spectra of Tween has not been found to interfere with the desired spectra of the chemicals deposited. It should be noted that to prevent blocking the fine holes in the print head, all printing liquids are filtered to 1.0 µm.

B.3.4 Printing Procedure

A number of tests are required to determine how various printer settings and chemical parameters affect the chemical mass deposited by the printer. Printer settings include resolution, drop size, drop volume, number of coats, maximum ink, and number of print cartridges used. Chemical parameters include viscosity, surface tension, and density. Chemicals may be liquid or a solid dissolved in suitable solvent. The volume per droplet may be specified in the printing program as 1.5, 3.0, 7.0, 14.0, or 21.0 pL; however, these volumes were determined using printer ink and are not necessarily accurate for the chemicals to be deposited. Determination of the actual chemical mass deposited by the printer may require a number of steps. The obvious procedure is to weigh the substrate before and after chemical deposition. In practice, this procedure is not always practical because the anticipated chemical mass may be in the milligram or microgram range, and the available substrate may be weighed in grams. In these cases, the resolution of the available laboratory scale may prove inadequate.

The procedure used for determining the chemical mass deposited on a heavy substrate is to initially use a small, lightweight aluminum substrate to determine the droplet volume. This may be accomplished by weighing the substrate before printing and again after the substrate is dried. With knowledge of the chemical concentration of the liquid, the droplet volume may be calculated. Some chemicals being printed, such as ammonium nitrate, are very hygroscopic and care must be exercised when weighing the substrate. Weight will initially decrease as the substrate dries. The weight will then increase as moisture from the air is absorbed by the chemical. The weight will eventually stabilize as the chemical moisture content approaches that of the air. This stabilized weight measurement probably introduces the least experimental error because it is assumed that the present chemical moisture content is similar to the moisture content when the chemical was first dissolved.

The following are useful chemical deposition formulas. The constant *K*, which is dimensionless, was derived to simplify calculations by allowing numerical values to be used for droplets in picoliters, area in square centimeters, and concentrations in grams per liter. The value of *K* is determined by droplet area, which varies with printer resolution

$$C = (S \times K)/P$$

$$P = (K \times M)/(A \times C)$$

$$M = (P \times A \times C)/K$$

$$S = M/A$$

where

A is surface area (cm^2);
P is droplet (pL);
C is liquid concentration (g/L);
K is 9.775E+6 (for 720 dpi), K is 2.44E+6 (for 1440 dpi);
M is mass deposited (g); and
S is surface concentration (g/cm^2).

B.3.5 Print Modes

To determine printer accuracy, various print mode settings were tested to evaluate the material concentrations on surfaces. Material densities were controlled by varying the volume of drops (picoliters per dot) and/or dots per inch settings of the printer. For this experiment, 32 g/L of potassium chlorate was printed on 2 × 2 in. pattern aluminum substrates. All the samples printed were heated to 80 °C. The printer setting combinations were 1.5, 3, 7, 14, and 21 pL and 720 × 720 and 1440 × 1440 dpi. All samples were weighed after deposition, and all printing was unidirectional.

B.3.5.1 Print Mode Files

The following print mode files were used, and the results are shown in Table B-2:

720 × 720 dpi, IR3, No ICC, 21 pL, uni
1440 × 1440 dpi, IR3, No ICC, 21 pL, uni

720 × 720 dpi, IR3, No ICC, 14 pL VDS1, uni
1440 × 1440 dpi, IR3, No ICC, 14 pL VDS1, uni

720 × 720 dpi, IR3, No ICC, 7 pL VDS2, uni
1440 × 1440 dpi, IR3, No ICC, 7 pL VDS2, uni

720 × 720 dpi, IR3, No ICC, 3 pL VDS3, uni
1440 × 1440 dpi, IR3, No ICC, 3 pL VDS3, uni

Table B-2. Print Mode vs Dot Size

Dot Size	VDS1 (pL)	VDS2 (pL)	VDS3 (pL)
Small	7	3	1.5
Medium	14	7	3
Large	21	14	7

B.3.5.2 Results

The following results were obtained during testing:

- It was determined that the variable dot size, “Small Dots” settings should not be used because the printer did not deposit consistent mass on the surface.
- 3pL setting: Did not print all the way when heated but worked fine without the heat. Assumption is that printer head holes were dried by the heat and clogged.
- 7 pL setting: Worked the best with VDS2, all medium dots.
- 14 pL setting: Worked the best with VDS1, all medium dots.
- 21 pL setting: Worked the best with VDS1, all large dots.

Actual picoliters per drop versus printer settings are shown in Table B-3. Results indicate that for accurate material deposition, printer calibration of drop volume is necessary.

Table B-3. Actual Picoliters per Drop

Dots per Inch Setting	Picoliters Setting	Measured Mass	Picoliters Measured	Error (%)
720 × 720	1.5	90 µg	1.07	–28.6
720 × 720	3	260 µg	3.08	+2.67
720 × 720	7	690 µg	8.17	+16.7
720 × 720	14	1.34 mg	15.86	+13.3
720 × 720	21	2.11 mg	24.97	+18.9
1440 × 1440	7	2.68 mg	7.93	+13.2
1440 × 1440	14	5.09 mg	15.06	+7.57
1440 × 1440 at 80 °C	21	7.74 mg	22.90	+9.05

B.3.6 Verification of Deposited Surface Concentration

The density of printed chemical deposition was verified using several methods including calculation, mass measurement, and chemical laboratory analysis. Two inch square patterns of potassium perchlorate were printed at 720 dpi using 14 pL per drop on 3 × 3 in. aluminum panels. The objective was a deposition of 50 µg/cm². Laboratory analysis of 16 samples showed that the average deviation from 50 µg/cm² was approximately 7%.

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